

# Thermal History of the Engine Oil and Its Effects on Low-Temperature Pumpability and Gelation Formation

Theodore W. Selby  
Savant, Inc.  
and  
Gregory C. Miiller  
Tannas Co.

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## ABSTRACT

Low-temperature engine oil pumpability has been a concern for OEMs, engine oil formulators, and additive manufacturers for a number of years particularly since a significant number of air-binding failures in 1980 and '81. On careful investigation of the cause of such field failures, it was found that oil sensitivity to a particular combination of weather conditions was responsible. The experience also suggested that many other low-temperature weather conditions might produce engine-damaging gelation. Thus, it seemed desirable to develop a bench test that would induce and measure gelation that might form in engine oil by continuously measuring slowly cooling oil over an extensive low-temperature range. This led to the development of the Scanning Brookfield Technique (SBT) first reported in 1982.

With the rapidly growing use of more highly paraffinic, but gelation-prone base oils as well as vegetable oils and fuels, the effects of these components of modern engine oils on oil gelation temperatures and severity are of interest. To effectively address this area of interest, this paper presents the background of the Scanning Brookfield Technique, its basis of developing measures of gelation phenomena called the Gelation Index and Gelation Index Temperature, and initial studies of the factors affecting the formation and growth of the gelation structure.

## BACKGROUND AND INTRODUCTION

The need to measure the low-temperature pumpability of engine oils has been clearly recognized since the 1970's [1-6]. This need was underscored by a number of engine failures occurring in the winter of 1980 [7] caused by the formation of a gelled condition in a popular engine oil (as well as engine failures in Europe the following winter of 1981).

The subsequent development of the Scanning Brookfield Technique (SBT) [8] was a direct response to these field occurrences.

**GELATION AND AIR-BINDING ENGINE FAILURE** - Earlier engine studies [9] conducted under a task group of the ASTM (Committee D02, Subcommittee 07) showed that a gelled structure which could develop under certain cooling conditions in the crankcase would only allow the oil immediately above the engine's oil pump screen to be pulled into the pump.

Figure 1 illustrates that as the oil pump draws a vacuum, atmospheric pressure forces a vertical column of the gelled oil into the oil pump, forming an air-filled vortex that will not close because of the structural stiffness of the surrounding gelled oil. Thus, after this relatively small amount of engine oil passes into the pump, only air will follow and, consequently, the oil pump becomes air-bound. Oil pressure drops to essentially zero. In this way, gelation was found to prevent oil circulation and cause starvation of those engine components depending on oil lubrication. (Such engine response produced by oil gelation had been anticipated in the early 1960s from studies of oil rheology at low temperatures and was termed 'air-binding' at that time [10].)

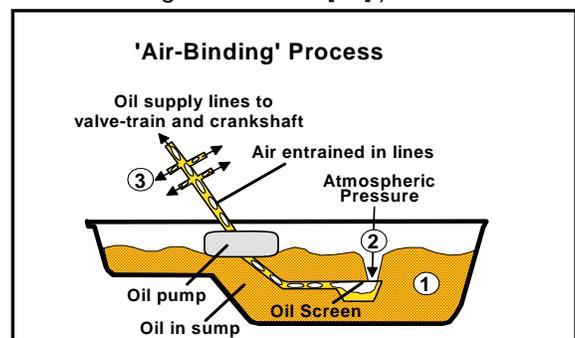


Fig. 1 – Diagram of 'air-binding' process – 1. Gelled engine oil, 2. atmospheric pressure forcing core of oil into screen followed by air, 3. air passing to lubrication sites.

**INDUCED GELATION FORMATION** – As will be shown in later sections of the paper, it is important to try to determine if, under any cooling condition in nature in which the automobile is used, gelation can form in engine oil at a level that would cause air-binding. The Scanning Brookfield Technique was developed to induce structure formation in gelation-susceptible engine oil and thus reveal if, under any circumstance, air-binding might occur.

The technique was based on an earlier observation in low-temperature capillary viscometry that latent gelation response of oil at low temperatures could often be induced by very slowly and laminarily shearing the fluid. It was reasoned that by gently causing oil molecules to slide past one another in laminar flow, those oil molecules capable of producing gelation would be physically moved into a proximity with one another that might not have otherwise occurred with Brownian motion – particularly at lower temperatures and relatively high viscosities where the natural motion of these larger molecules is considerably retarded. [11].

**THE SCANNING BROOKFIELD TECHNIQUE** – There are several important steps in the Scanning Brookfield Technique.

**Set-up, Pre-Heat, and Cooling** – Although a liquid bath was not used in the studies reported in this paper, Figure 2 is helpful in showing SBT viscometer heads with attached rotor/stator cells (indicated by arrow) immersed in a liquid bath in preparation for test.



Fig. 2 – TAV-3 viscometer heads set up for SBT analysis on a liquid bath.

Radial clearance between the rotors and stators in the viscometer cells is approximately 1.5 millimeter. The empty test cell stator is filled to the proper level with the test fluid and placed in either a water bath or oven held at 90°C for 90 minutes to remove the oil's 'memory' (discussed more fully below).

Following this 'memory'-removing, pre-heating step, the rotor is placed in the stator and both are coupled to the viscometer head. The rotor/stator cell and test fluid are then inserted into the temperature-controlling bath which is usually (but not necessarily) initially held at -5°C. After the cell and fluid have cooled to this temperature, and following the procedure of ASTM Method D5133 [12], the rotor is set to turn at 0.3 RPM and a programmed sample cooling rate of 1°C per hour is initiated. It is necessary to continuously record the torque/viscosity information from the calibrated viscometer head in order to determine the severity of gelation and the values of the Gelation Index and Gelation Index Temperature. Greater detail is given in the ASTM Method and in previous papers [13,14].

**EXAMPLE OF SBT ANALYSES** – Figures 3 and 4 show responses of two oils, one with, and one without, evident gelation. Figure 3 shows the smooth, exponential viscosity-temperature curve of a non-gelating oil contrasted to a gelating oil which begins structure formation at about -14°C.

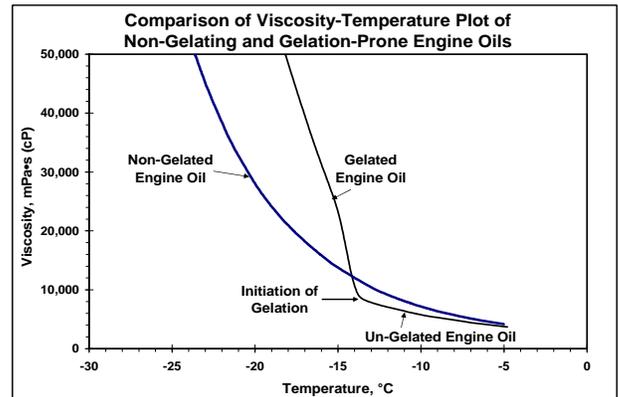


Fig. 3 – SBT viscosity-temperature scans of two oils of which one shows gelation.

Figure 4 shows analyses of the viscosity-temperature curves of Figure 3 using the empirical MacCoull, Walther, Wright equation [15,16,17]. This equation gives a straight line having negative slope when applied to a smooth, exponential viscosity-temperature curve. Taking the first derivative of this linear equation gives an essentially flat, horizontal line as in Figure 4. However, as also shown in Figure 4, applying the same technique to the viscosity-temperature curve of the gelating oil, yields a curve with a Gelation Index of 22 at -14.3°C Gelation Index Temperature (again, for further detail on this procedure, see References 11 and 14).

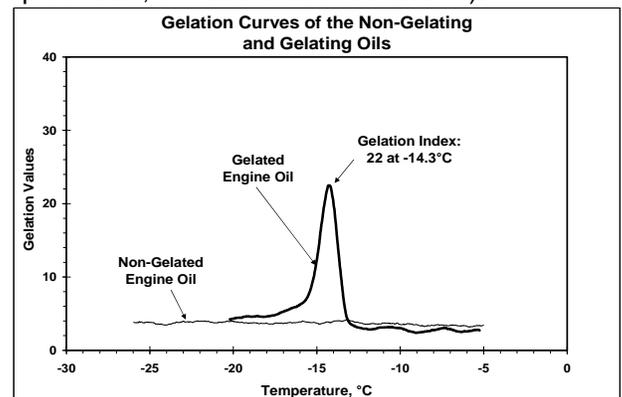


Fig. 4 – Gelation Index data generated from the viscosity-temperature curves of the oils shown in Figure 3.

**SIGNIFICANCE OF INFORMATION FROM THE GELATION INDEX** - It is evident from Figure 3 that the process of gelation formation shows itself clearly by its effect on the viscometric response of the oil. That is, as gelation forms, the developing structure impedes the flow of the remaining oil and viscosity increases even more rapidly than the exponential rate normally associated with decreasing temperature.

The amount of gelation developed is also shown by how slowly the oil's viscosity-temperature curve returns to normal exponential behavior at which point evidence of structural formation ceases. Rate of structural formation starts to decrease after the inflection point of the viscosity-temperature curve for the gelating oil in Figure 3 which is also associated with the Gelation Index peak and Gelation Index Temperature shown in Figure 4.

From these observations, it is reasonable that the steeper the departure of the viscosity-temperature relationship from the initially normal exponential form typical of non-gelating behavior, the more rapidly structure is forming. Similarly, the larger the Gelation Index value becomes, the more structure formation that has occurred. Lastly, return to exponential viscosity-temperature behavior indicates the exhaustion of gelating components of the oil under the test conditions.

The Scanning Brookfield Technique has been used for a number of years to categorize the low-temperature pumpability of engine oils. In such use, it has played a major role in showing better ways of controlling gelation and air-binding – arguably the most damaging and least predictable form of pumpability problems.

## **GELATION MEASUREMENT OF MODERN OILS**

Today, rapidly increasing use of mineral base oils having considerably higher paraffinic content than past solvent-refined base oils – as well as the use of bio-lubes and bio-fuels with their inherently higher low-temperature gelation properties – calls for careful identification of gelation tendency. This is especially the case for engine oils marketed in climates that can experience cooler temperatures occasionally or frequently.

In this regard, another area of importance is to clearly understand how best to measure the response of modern, highly paraffinic base oils that require more precise treatment with pour-point depressants to avoid air-binding. Such base oils are individually highly sensitive to the proper level of pour-point depressants.

Since the Scanning Brookfield Technique is similarly sensitive to the formation of gelation, the critical borderline Gelation Index zone of 12 to 15 has been of interest in further improvement of the method and instrument. (This borderline zone is not arbitrarily chosen since one of the field-failing oils in 1980 had a Gelation Index level of 16.)

**EFFECTS OF COOLING FROM PRE-HEATING CONDITIONS** - In the course of this study, it was considered important to determine the role of test sample cooling from the pre-heating temperature of 90°C required for removal of the oil's 'memory' of prior exposure to gelating conditions.

**DESCRIPTION OF OIL 'MEMORY'** – Oil 'memory' is an anthropomorphic term indicating that oil can carry residual behavior resulting from its previous combined thermal and rheological history which will affect its further behavior. This term was generated by early studies of gelation when it became evident that an SBT

test for gelation would not give the same results if a sample were simply analyzed again over the same temperature range. In such case, the second analysis had much less evidence of gelation-forming tendencies over the same temperature range.

To obtain repeatable results with the same sample in the glass stator, it was found necessary to pre-heat the sample to about 90°C for 1½ hours prior to analysis. Using pre-heat, results became very repeatable in both the value of Gelation Index shown and the temperature at which gelation began to form.

**Explanation of Oil 'Memory'** – A reasonable explanation of oil's 'memory' and the need for pre-heating the sample is that the structure formed in oil gelation at lower temperatures is not easily disassociated at temperatures up to and beyond ambient.

Such disassociation to essentially individual molecules can only occur at temperatures imparting sufficient energy to those molecules forming the structure at lower temperatures to completely break up the molecular association originally leading to structure formation in the oil at lower temperatures. Unless this occurs, at least some of the associated molecules involved in forming parts of the previous gelled structure are no longer available to the same extent in rebuilding the gelled mass in the oil. As a consequence, when a second analysis of the same sample is made without pre-heating and the resulting 'memory erasure', the Gelation Index generated will be considerably lower.

**Solute/Solvent/Miscibility Relationship** – Part of a reasonable explanation of the difficulty of removing the 'memory' of oils having gelation tendencies is to consider that the various components of a mineral oil have different degrees of solubility and/or miscibility in one another depending on the temperature at which they are in association and their molecular configuration. Depending on such factors as its molecular weight and crystallization temperature, at some higher temperature each hydrocarbon molecule passes from a solid-but-soluble state to a liquid-and-miscible state. Obviously, at sufficiently high temperatures, most if not all of the hydrocarbon molecules will be mutually miscible.

As temperature of this hydrocarbon mixture is lowered, more and more molecules will pass from the miscible state and enter the soluble state until the remaining liquid hydrocarbons will no longer have sufficient solvency to prevent some of the less soluble hydrocarbon molecules from leaving solution – first leading to the so-called Cloud-Point and below this temperature to the Pour Point. Even before the Cloud Point occurs, however, it is not unreasonable to consider that, under quiescent and slow-cooling conditions; some of these molecules would form associations and three-dimensional networks within the disordered mass of the more fluid components.

In such a slow-cooling scenario, the combination of 1) the growing numbers of molecules leaving the fluid state and entering the soluble state, 2) exponentially increasing viscosity of the mixture, 3) increasing

numbers of molecules whose Brownian motion is slowed sufficiently to encourage linkage in the nascent structure, are all component pieces of the resulting gelation that at lower temperatures would cause air-binding and engine failure.

**SUPPORTING EVIDENCE FROM FIELD FAILURES –** Evidence for such an interrelationship between the temperature of incipient formation of gelation structure and the ultimate air-binding failure temperature was clearly shown to be the cause of the 1980 pumpability failures through a seminal engine cold-room study in 1981 by Stambaugh and O'Mara (7) using temperature-cycles similar to those that had produced the gelated, air-binding field failures.

The gelation-inducing mechanism, by which the engine oil failed, was very precisely orchestrated by the weather in that the temperature hovered at a specific, moderately low, temperature for about seven or eight hours and then dropped steadily to a temperature a minimum of several degrees lower. Only then would engine-failing air-binding occur.

Interestingly and importantly from the viewpoint of understanding gelation mechanisms, the cold-room temperature at which the oil in the engine was 'soaked' had to be precisely held at  $-9\pm 0.5^{\circ}\text{C}$  ( $16\pm 1^{\circ}\text{F}$ ) for eight hours and then be lowered slowly over another eight hours by at least  $5^{\circ}\text{C}$ . If either of these conditions were not met, air-binding would not occur.

It seems clear that the initial 'soak' enabled the development of the incipient gelation structure during which those molecules capable of participating had sufficient time and opportunity to migrate to the structure formation site(s) and were at a temperature where they were just slow enough to readily become a part of the structure formation (note the very narrow temperature window within which structure could initiate).

After sufficient time elapsed for adequate structure to be formed, a further  $5^{\circ}\text{C}$  (minimum) decrease in temperature was necessary to provide the effects of further structure and/or significantly lower levels of flow of the remaining fluid portion of the oil enmeshed in the interstices of the structure.

From the foregoing and the principles behind the development of the Scanning Brookfield Technique it was not surprising that the SBT also showed the formation of gelation in this failing oil at  $-9^{\circ}\text{C}$  [8].

It was later found that the  $5^{\circ}\text{C}$  minimum difference between evidence of gelation formation in the SBT test and the actual occurrence of air-binding [14] gave good correlation with less sharply temperature-sensitive reference oils in the ASTM cold-room engine tests [9].

With this evidence of gelation precursor formation, it was of interest to determine to what degree and at what temperatures such precursor formation takes place and initial studies are reported in this paper.

## FURTHER STUDIES OF GELATION

REFERENCE OILS - Three reference oils were used:

Reference Oil LNP-5 – One of these was the Newtonian oil, LNP-5, used for calibrating the Tannas viscometer heads applied in SBT analysis.

Reference Oil GIR-200 – The second oil was a non-Newtonian oil, GIR-200, provided by the Tannas Co. for its equipment. This oil has a  $35.1\pm 5.6$  Gelation Index and a  $-12.0\pm 1.5^{\circ}\text{C}$  Gelation Index Temperature.

Reference Oil GIR-150 – The third oil, GIR-150, was also a non-Newtonian oil provided by the Tannas Co., and has a similarly well established  $20.1\pm 3.2$  Gelation Index at  $-13.7\pm 2.0^{\circ}\text{C}$  Gelation Index Temperature.

INSTRUMENTATION – Instruments used were:

Viscometer Heads – Two Tannas Model TAV-3 viscometer heads (see Figure 2) were used. These are capable of recording up to approximately 100,000 mPa•s (cP) before exceeding torque limits of the viscometer.

Viscometer Cooling – All new work reported in this paper was conducted in a specially designed, directly refrigerated console containing stainless steel stators instead of the glass stators used in liquid baths. (This cooling approach was called the 'dry-bath' approach since it eliminated the use of a liquid bath.) The instrument was designed so that two viscometers could be run simultaneously. Temperature control of the dry-bath is programmable and the torque data from each head are recorded automatically using the program developed earlier for liquid-bath ASTM analyses.

METHOD – ASTM Method D5133 [12] was used in these studies after initiation of the  $1^{\circ}\text{C}/\text{hour}$  cooling procedure at  $-5^{\circ}\text{C}$ . However, different cooling rates from the pre-heat temperature of  $90^{\circ}\text{C}$  were applied in studies of the influence of cooling rate and other conditions on the formation of gelation.

Pre-Heating and Subsequent Cooling Methods – Four methods were applied to pre-heating and subsequent cooling to the  $-5^{\circ}\text{C}$  temperature preparatory for starting SBT analysis. In none of the cooling cycles was the cooling rate linear:

1. The sample-containing stators were heated in the dry bath to  $90^{\circ}\text{C}$  for  $90\pm 2$  minutes then brought to  $-5^{\circ}\text{C}$  in a cooling time of 45 minutes at which point the rotor (fixed to the viscometer) was lowered into the stator and the SBT D5133 test started at 0.3 RPM when the dry bath was again at  $-5^{\circ}\text{C}$ .
2. The sample-containing stators were heated at  $90^{\circ}\text{C}$  in a water bath and removed after  $90\pm 2$  minutes. At this point the stators were allowed to cool for precise but variable time intervals. Following this, the rotor (fixed to the viscometer) was lowered into the stator and both inserted in the dry bath being held at  $-5^{\circ}\text{C}$ . The SBT D5133 test was started when the dry bath returned to  $-5^{\circ}\text{C}$ .

- The stators, samples, and rotors were heated in the dry bath to 90°C for 90±2 minutes while the rotor was turning at either 0.3 or 12 RPM both during the pre-heating period and the cooling time until the dry bath was at -5°C at which point the SBT analysis was started at a rotor speed of 0.3 RPM.
- The stators, samples, and rotors were heated in the dry bath to 90°C for 90±2 minutes after which the refrigeration was initiated without any motion of the rotor until the dry bath was at -5°C at which point the SBT analysis was started.

**CALIBRATION OF THE VISCOMETER HEADS** – The two viscometer heads and their associated rotor-stator cells were calibrated by determining the torque output of the viscometer heads when running the Newtonian Reference Oil LNP-5 over the range of -5°C to -40°C. Torque and temperature data collected by the SBT program on a computer gave the slopes and intercepts used to determine viscosity and Gelation Indices. Torque-viscosity correlation was acceptable (Correlation Coefficient R > 0.999) according to ASTM D5133.

## RESULTS

**GIR 200** – Of the two reference gelating oils, GIR 200 had the highest level of gelation at a Gelation Index of about 35 at -11° to -12°C. It was considered that of the two reference oils, gelation with GIR 200 would be least repressed by pre-heat cooling conditions.

Methods 1 and 2 were the cooling approaches used, and results are shown in Table 1.

Viscometer	Sample Cooling Time	Gelation Index	Gelation Index Temperature	Comment
	Minutes		°C	
1	45	35.0	-11.4	Cooling Method 1
2		37.5	-11.2	
1	40	31.0	-11.9	Cooling Method 2
2		35.7	-11.5	
1	30	33.8	-11.6	Cooling Method 2
2		36.4	-11.2	
1	30	32.2	-11.8	Rerun Method 2
2		34.2	-11.5	
1	10	34.8	-11.7	Cooling Method 2
2		35.7	-11.3	

It is evident that stator cooling time has essentially no evident effect on gelation. Moreover, the Gelation Index and Gelation Index Temperature values correspond well with expected values previously given for GIR 200.

Repeatability was shown both by values of Gelation Index and Gelation Index Temperature as well as in the two sets of data obtained at 30-minute cooling time indicating good repeatability from instrument to instrument. These results on GIR 200 suggest that, for its pattern of gelation, structure forms only after starting

cooling at -5°C and inducing the laminar flow of the oil provided by the slowly turning rotor in the sample.

**GIR 150** – The first study on GIR 150 applied Method 2 for 50, 20, and 10 minutes cooling before insertion of the rotor-stator cell in the dry-bath. Since the Gelation Index level of about 20 shown by this reference oil was less developed than the level of 30 shown by GIR 200, it was anticipated that gelation formation in GIR 150 might be more sensitive to stator cooling conditions.

This was shown to be the case in the first run on GIR 150 with a 50-minute stator cooling period from the 90-minute, 90°C preheat. As evident in Table 2, results for both viscometers showed that no gelation was generated during the ASTM D5133 analysis which followed the cooling.

Viscometer	Sample Cooling Time	Gelation Index	Gelation Index Temperature	Comment
	Minutes		°C	
1	50	5.5	-22.7	Cooling Method 2
2		5.2	-23.4	
1	20	16.9	-13.8	Cooling Method 2
2		16.4	-13.8	
1	10	19.0	-13.7	Cooling Method 2
2		19.8	-13.6	

This could be readily explained by the hypothesis that:

- Most of the gelation-forming molecules had been employed in structure-building during the 50-minute cooling period, and
- The somewhat fragile structure was torn into shards early in the D5133 analysis.

Shorter stator cooling periods of 20 and 10 minutes were immediately run to test this hypothesis. As shown in Table 2, they seemed to confirm it with progressively higher Gelation Index levels at the Gelation Index Temperature values of approximately -14°C expected. In fact the 10-minute cooling time seemed to be brief enough to preserve most of the structure-building molecules in a suspended state until the SBT analysis was run. This latter test gave results normally expected from GIR 150 as previously noted in the information on the Reference Oils.

Fortunately, these findings support the directives generated several years ago in ASTM Method D5133 to transfer the pre-heated sample to the cold bath for testing as quickly after pre-heating,

**IN-BLOCK COOLING OF GIR 150** – The study of GIR 150 was continued in order to gain further insights into the gelation structure-forming process. It was thought interesting to conduct the pre-heating step in the dry-block instrument which would permit conducting the entire Scanning Brookfield Technique within the dry-block. Techniques 1, 3, and 4 were applied using no motion of the rotor in the stator or either 0.3 or 12 RPM.

Table 3 shows both the earlier data of Table 2 (the first six rows) plus eight rows of data obtained from Methods 1, 3, and 4 and it is of interest to compare results.

Table 3 - Experiments With Stator Cooling in Block Compared to Stator Cooling Out of Block Regarding Gelation Response of GIR 150				
Viscometer	Sample Cooling Time	Gelation Index	Gelation Index	Comment
	Minutes		Temperature °C	
1	50	5.5	-22.7	Cooling Method 2
2		5.2	-23.4	
1	20	16.9	-13.8	Cooling Method 2
2		16.4	-13.8	
1	10	19.0	-13.7	Cooling Method 2
2		19.8	-13.6	
1	45	10.0	-13.7	Cooling Method 1
2		9.8	-13.9	
1	45	16.2	-13.8	Method 3 0.3 RPM
2		16.6	-13.8	
1	45	18.6	-13.7	Method 3 12 RPM
2		18.4	-13.6	
1	45	18.7	-13.7	Cooling Method 4
2		18.1	-13.7	

**SAMPLE PRE-HEATING AND COOLING WITHOUT ROTOR** – Methods 1 and 2 are somewhat similar with the exception that with Method 2 the GIR 150 is pre-heated and cooled outside of the dry-bath. In contrast, with Method 1 GIR 150 is pre-heated inside of the dry bath and cooling takes a slightly shorter period of time. In both approaches, the viscometer head and rotor are placed on and in the stator, respectively, just before the ASTM D5133 test begins.

Interestingly however, this five minute shorter period of cooling time for Method 1 does seem to prevent loss of all gelation-formation that is shown by Method 2. Not only does the Gelation Index increase from about 5 to approximately 10 but the Gelation Index Temperatures assume their expected values of about 14°C.

**PRE-HEATING AND LAMINAR FLUID MOTION DURING COOLING** – On the basis of the previous experiments, it was considered that if the nascent structure of GIR 150 formed during quiescent cooling, moving the fluid lamilarly with the rotor might have either or both of two effects:

1. To prevent the nascent structure from forming, or
2. To, at some temperature, help the nascent structure to form and, if so,
3. Any difference might be found to be dependent on rotor speed.

Results shown with Method 3 using in-block cooling give an answer. The rotating speed of 0.3 RPM applied during the cooling period does have an evident effect in preventing pre-formation of much – but not all – of the gelation structure before the ASTM D5133 analysis is run. At this rate of rotation, the Gelation Index that occurs later during the analysis is found to be about 16 to 17 and occurs at the expected Gelation Index Temperature -13.8°C.

When the results of 0.3 RPM are compared to the results at 12 RPM, it seems clear that although this 0.3 RPM speed does seem to inhibit precursor gelation

formation, it is not as effective as 12 RPM. The latter speed seems to totally inhibit precursor formation as judged by the Gelation Index values.

Pre-Heating in Instrument with No Rotor Motion During Heating or Cooling – However, most interestingly, using Method 4 in which the rotor is simply placing in position as the sample of GIR 150 in the cell is first pre-heated and then cooled to -5°C in 45 minutes, shows essentially full gelation response. That is, the presence of the rotor in the stator in some manner inhibits precursor formation of gelation tendencies as effectively as turning the rotor at 12 RPM.

This data suggests that the presence of the rotor in the stator interferes with the migration of the molecules forming the nascent structure by forcing the sample into a narrow 1.8 mm thick by 18.4 mm inner diameter by 85.5 mm long torus between the rotor and the stator. Migration of such molecules would be significantly impeded. From this point of view the results of applying Method 3 at 0.3 RPM may have been an indication of partially helping formation of the nascent structure.

## DISCUSSION AND CONCLUSIONS

**UNDERSTANDING GELATION OF ENGINE OIL** – Although no sudden incidents as severe and evident as those in 1980 and 1981 have been recorded since that time, this, of course, does not mean that air-binding is an occurrence of the past. With hope, the instruments, methods and specifications developed as a consequence of that experience have held the phenomenon at bay – at least in those areas of the world in which such measurements are made.

However, with the rapid growth in use of more highly paraffinic base oils and the use of vegetable oils and fuels in diesel engines, the degree of vulnerability to incidents of air-binding is growing – particularly in areas of the world outside of North America and Japan (where engine oils must meet ILSAC low-temperature pumpability specifications which include the SBT). Unfortunately, it is difficult to distinguish an engine that has failed by air-binding of the oil and one that has failed through some other problem and this has been one of the OEMs serious concerns.

The evident answer to the need to protect engines against the already obviously unpredictable effects of nature is to understand more about the formation of gelled structures in this period of rapid change of base oils into more gelation-sensitive types.

**INCIPIENT GELATION AT AMBIENT AND HIGHER TEMPERATURES** – Results of this study indicate that the phenomenon of engine oil gelation may also be initiated at ambient or higher temperatures and not simply be restricted in all cases to formation at lower temperatures. In fact, the data suggest that sufficient, but comparatively fragile, gelation can form at ambient and somewhat higher temperatures that, when broken up in first rotor movement, may preclude development of an otherwise failing level of Gelation Index at lower temperatures.

The present study seemed to indicate that inhibiting migration of structure-forming molecules until the temperature and viscous conditions of the engine oil are appropriate is a means of insuring obtaining meaningful and reasonably repeatable results from the SBT.

It has also been demonstrated that after pre-heating (if preheating is done in an independent device such as an oven), it is imperative to quickly set up the viscometer-cell configuration and move the cell to the location at which the analysis will be made to avoid any incipient structural formation that would reduce the Gelation Index value.

The effect of applying rotor movement during sample cool-down from pre-heating suggests that if the rotation speed is relatively slow, the formation of precursor structures can be simultaneously damaged by the effect of fluid motion but also encouraged by the rotor-assisted molecular migration. At higher rotor speeds structure damage seems to overwhelm structure formation.

Differences in the responses of GIR 150 and 200 also indicate that gelation formation temperatures are dependent on the individual formulations of the lubricant.

A previous study of diesel engine oils suggested that considerable gelation may form above -5°C [18,19] and this present study further supports those observations.

**GELATION SENSITIVITY TO COOLING TIME AFTER PRE-HEAT** – The results of this study also suggest that Gelation Indices of 20 or less may be much more sensitive to the handling of the sample before analysis. GIR 200 at a Gelation Index level of approximately 35 was much less, if at all, affected by cooling rate compared to GIR 150. On the other hand, this may be a function of the composition of the base stock and additives – comparison of these two oils is interesting but certainly not conclusive.

An alternative suggestion regarding structure formation after pre-heating involves the migration time required for the pour-point depressant to become effective in preventing structure formation during cooling after pre-heating has been suggested by Reviewer 4 (anonymous) of this paper and this scenario also has merit

It should be mentioned that engine operating conditions are most often quite enough to remove all memory of the engine oil before the engine is turned off to sit quietly while weather conditions cools the oil to produce whatever form of response the oil will exhibit.

## SUMMARY

In presenting the data and observations in this paper, the authors wish to clearly point out that these data are the results of initial studies and that further work along these and other lines of interest are underway to verify and extend these observations. Nonetheless, many of the observations made are mutually consistent and, as such, hold promise of further understanding of the process and control of gelation and air-binding.

## ACKNOWLEDGEMENTS

The authors would like to express appreciation of the contributors to the application and study of gelation using the Scanning Brookfield Technique. A number of these individuals have also carried this method into other areas of fuels and lubricants with considerable success. The fact that much of this work is proprietary and thus has not yet been published emphasizes its value to those practicing the technique.

## BIBLIOGRAPHY

1. Stewart, R.M and Selby, T.W., "The Relationship between Oil Viscosity and Engine Performance – A Literature Search", *SAE International Automotive Congress and Exposition*, Detroit, 1977. Published in *The Relationship between Engine Oil Viscosity and Engine Performance – Part I*, A joint publication of the SAE and ASTM, ed. Stewart, R.M. and Selby, T.W., SAE SP 416 and ASTM STP 621, pp. 1-20, SAE, Warrendale, PA., 1977.
2. McMillan, M.L., "Engine Oil Viscosity Classification – Past, Present and Future", *ibid.*, pp. 21-32, 1977.
3. Stewart, R.M., "Engine Oil Pumpability and Crankability Tests on Commercial "W" Graded Engine Oils Compared to Bench Test Results", Published in *The Relationship between Engine Oil Viscosity and Engine Performance – Part III*, A joint publication of the SAE and ASTM, ed. Stewart, R.M. and Selby, T.W., SAE SP 429 and ASTM STP 621, pp. 1-14, SAE, Warrendale, PA., 1978.
4. Eberan-Eberhorst, C.G.A., et.al., "Low Temperature Starting and Pumpability Requirements of European Automotive Engines", Published in *The Relationship between Engine Oil Viscosity and Engine Performance – Part V*, A joint publication of the SAE and ASTM, ed. Stewart, R.M. and Selby, T.W., SAE SP 460 and ASTM STP 621-S2, pp. 27-52, SAE, Warrendale, PA., 1980.
5. Stover, W.H., "Cold Starting and Oil Pumpability – An Evaluation of New and Used Oils in Gasoline Engines", *ibid.*, pp. 71-84, 1980.
6. Shaub, H., et.al., "Predicting Low Temperature Engine Oil Pumpability with the Mini-Rotary Viscometer", *ibid.*, pp. 107-126, 1980.
7. Stambaugh, R.L. and O'Mara, J.H., "Low Temperature Flow Properties of Engine Oils", *SAE International Automotive Congress and Exposition*, Detroit, MI, Paper 820509, SAE, Warrendale, PA, Feb. 1982.
8. Selby, T.W., Presented formal discussion of Ref. 7, Feb. 1982. Included in ASTM Research Report D02-1448 for ASTM Method D5133, 1990.
9. Low-Temperature Pumpability Characteristics of Engine Oils in Full-Scale Engines, *ASTM Data Series Publication 57*, Prepared by the Data Analysis Panel of ASTM D-2, R&D Division VII-C on Non-Newtonian Low-Shear Phenomena, ASTM,

10. Selby, T.W., "Viscosity and the Cranking Resistance of Engines at Low Temperatures", *Sixth World Petroleum Conference*, Section VI, Paper 17, Frankfurt/Main, June 1963.
11. Selby, T.W., "Pumpability – Past Accomplishments, Present and Future Challenges", *Oil Flow Studies at Low Temperatures in Modern Engines*, ed. Shaub H., ASTM STP 1388, pp. 99-121, ASTM, West Conshohocken, PA, 2000.
12. ASTM Method D5133-05: Low Temperature, Low Shear Rate, Viscosity-Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique, *2008 Annual Book of ASTM Standards – Section Five*, pp. 950-961, ASTM, West Conshohocken, PA, 2008.
13. Shaub, H., "A History of ASTM Accomplishments in Low Temperature Engine Oil Rheology – 1966-1991", *Low Temperature Lubricant Rheology Measurement and Relevance to Engine Operation*, ed. Rhodes, R.B., ASTM STP 1143, pp. 1-19, ASTM, West Conshohocken, PA,, 1992.
14. Selby, T.W. "The Scanning Brookfield Technique of Low-Temperature, Low-Shear Rheology – Its Inception, Development, and Application", *ibid.*, pp. 33-64, 1992.
15. MacCoull, N., *Lubrication*, p.85, Texas Co., N.Y., NY, 1921.
16. Walther, C., *Erdöl und Teer*, Vol 4, p. 510, 1928.
17. Wright, W.A., "An Improved Viscosity-Temperature Chart for Hydrocarbons", *Journal of Materials*, Vol. 4, No. 1, p. 19, 1969. Wright.
18. McGeehan, J.A., et.al., "The Pivotal Role of Crankcase Oil in Preventing Soot Wear and Extending Filter Life in Low Emission Diesel Engines", *SAE International Automotive Congress and Exposition*, Dearborn, MI, Paper #1999-01-1525, SAE, Warrendale, PA, Feb. 1999.
19. Selby, T.W. and McGeehan, J.A., "Low Temperature Rheology of Soot-Laden, Heavy-Duty Engine Oils Using the Scanning Brookfield Technique", *SAE International Automotive Congress and Exposition*, Detroit, MI, Paper #2006-01-3352, SAE, Warrendale, PA, Feb. 1982.